

(12) AUSTRALIAN PATENT ABSTRACT

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(54) ADHESIVE PACK

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FUES

(74) SF

(57) Claim

17. The use of emulsion polymers adjusted to an ointment- or paste-like consistency which, although soft-spreading, will not flow in the absence of pressure and which contain an adhesive-type homo- and/or copolymer stabilized with thickeners as disperse solid in aqueous phase, in handle-like plastic containers with a headpiece, comprising an outlet opening, a pressure-controlled dispenser operable by the holding hand and a distributing element for the dispensed part of the emulsion polymer, as a ready-to-use adhesive pack held in one hand.

1. A ready-to-use adhesive pack, characterized by the following features in combination:

a) an adhesive based on emulsion polymers thickened to an ointment- or paste-like consistency which, although soft-spreading, will not flow in the absence of pressure

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(11) AU-A-45520/85

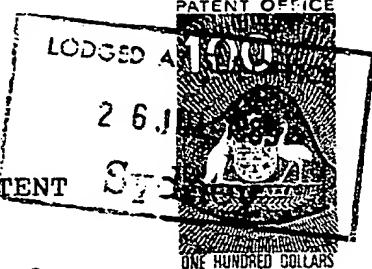
-2-

- and which contain an adhesive-type homo- and/or copolymer stabilized with protective colloids or thickeners as disperse solid in aqueous phase,
- b) a hand-held container accommodating the adhesive with an outlet opening for the adhesive and a device for the measured release thereof,
  - c) a device which reduces the adhesive-filled interior of the container in volume commensurate with the release of the adhesive in such a way that the adhesive remaining in the container is always bubble-free and safe against the re-entry of air.

SPRUSON & FERGUSON

COMMONWEALTH OF AUSTRALIA  
PATENTS ACT 1952

CONVENTION APPLICATION FOR A STANDARD PATENT



We HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN of  
Henkelstrasse 67, Dusseldorf, Federal Republic of Germany hereby  
apply for the grant of a standard patent for an invention  
entitled:

"A READY-TO-USE ADHESIVE PACK"

which is described in the accompanying complete specification.

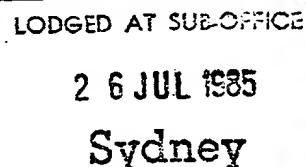
DETAILS OF BASIC APPLICATIONS

Number(s) of Basic Application(s):-

P 34 27 728.5, P 34 27 727.7, P 34 28 915.1,  
P 34 46 697.5 and P 35 22 363.4

Name of Convention Country in which Basic  
Application(s) were filed:-

Federal Republic of Germany

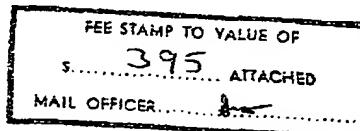


Date(s) of Basic application(s):-

27 July 1984, 27 July 1984, 6 August 1984,  
21 December 1984 and 22 June 1985 (respectively)

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DATED this SEVENTEENTH day of JULY 1985

HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN

By:

Registered Patent Attorney.

TO: THE COMMISSIONER OF PATENTS  
AUSTRALIA

SBR:ep 64W



**DECLARATION IN SUPPORT OF A CONVENTION  
APPLICATION FOR A PATENT OR PATENT OF ADDITION**

In support of the Convention Application made for a

patent

~~XPERKOMMANDITG~~

for an invention entitled

D-7M2/45/50/7251/7090-AU

"A READY-TO-USE ADHESIVE PACK"

Full name and ad-  
dress of Declarant.

I, Dr. Günter Schenck  
of Am Bühl 49  
4330 Mülheim / Germany

do solemnly and sincerely declare as follows:-

1. XXXXXXXX~~XPERKOMMANDITG~~ XPERKOMMANDITG

(or, in the case of an application by a body corporate)

1. I am authorised by HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN

the applicant for the ~~XPERKOMMANDITG~~ patent to make this declaration on its behalf.

Insert country and  
date of basic appli-  
cation and name of  
foreign applicant.

2. The basic applications as defined by Section 141 of the Act were made in  
the Federal Republic of Germany

on the

27th	day of	Juli 1984	all by
27th		Juli 1984	
6th		August 1984	
21st		December 1984	
and 22nd		June 1985	

Henkel Kommanditgesellschaft auf Aktien

XXXXXXXXXXXXXXXXXXXXXX~~XPERKOMMANDITG~~ XXXXXXXX~~XPERKOMMANDITG~~ XXXXXXXX~~XPERKOMMANDITG~~  
(or where a person other than the inventor is the applicant)

Full name and ad-  
dress of Inventor(s)

3. of Dr. Wolfgang Ritter, of Hochdahler Straße 32, 4010 Hilden, Germany  
 Dr. Hans-Christof Wilk, of An der Obererft 94, 4040 Neuss, Germany  
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 Dr. Wolfgang Dierichs, of Im Diepental 11, 4000 Düsseldorf 13, Germany  
 Dr. Günter Schenck, of Am Bühl 49, 4330 Mülheim/Ruhr, Germany and  
 Dr. Johann Friedrich Fues, of Otto Hahn Straße 157, 4000 Düsseldorf, Germany

are ~~X~~ the actual inventors of the invention and the facts upon which the applicant  
~~is~~ entitled to make the application are as follows:

The said applicant is the assignee of the  
actual inventors.

4. The basic applications referred to in paragraph 2 of this Declaration  
~~were~~ was the first applications made in a Convention country in respect  
of the inventions the subject of the application.

Declared at Düsseldorf this 24th day of June 1985

Signature of Declarant

To:

COMMONWEALTH OF AUSTRALIA  
PATENTS ACT 1952

COMPLETE SPECIFICATION

(ORIGINAL)

FOR OFFICE USE:

Class Int. Class

45520/85

Complete Specification Lodged:

Accepted:

Published:

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Related Art:

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Name of Applicant: HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN

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Federal Republic of Germany

Actual Inventor(s): WOLFGANG RITTER, HANS-CHRISTOF WILK,  
HANS-PETER HANDWERK, WOLFGANG DIERICHES,  
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Complete Specification for the invention entitled:

"A READY-TO-USE ADHESIVE PACK"

The following statement is a full description of this invention,  
including the best method of performing it known to us

SBR:ep 64W

ABSTRACT

The invention relates to new ready-to-use adhesive packs which are characterized by the following features in combination:

- a) adhesives based on emulsion polymers thickened to an ointment- or paste-like consistency which, although soft-spreading, will not flow in the absence of pressure and which contain an adhesive-type homo- and/or copolymer stabilized with protective colloids or thickeners as disperse solid in aqueous phase,
- b) containers held in one hand with an outlet opening for the adhesive and a device for the measured release thereof,
- c) a device which reduces the adhesive-filled interior of the container in volume commensurate with the release of the adhesive in such a way that the adhesive remaining in the container is always bubble-free and safe against the re-entry of air.

The preferred adhesives are stabilized aqueous emulsion polymers having viscosities (Brookfield RVT, 20°C/20 r.p.m.) of at least 15,000 mPas and more especially in the range from 50,000 to 500,000 mPas and contain polyethylene glycol or mixed polyethers of ethylene oxide and propylene oxide having an average molecular weight of at least 3000 as thickeners having virtually unlimited stability in storage.

## 1 A READY-TO-USE ADHESIVE PACK

There have been numerous proposals for adhesives and easy-to-handle adhesive packs which are used on an everyday basis in the office or home and also in workshops, for  
5 industrial purposes and in the DIY field.

Fluid, dissolved and/or emulsified or dispersed adhesives are particularly important. Known adhesives of this type include liquid adhesives based on solutions of a high molecular weight adhesive in solvents and free-flowing  
10 plastics dispersions or emulsions in which the adhesive-type polymer is present as disperse phase in a continuous liquid phase. These dispersion or emulsion adhesives may have an aqueous base. The fact that they can still be used after prolonged storage or after only occasional, portioned release  
15 of limited quantities is attributable above all to the ability of these fluid products to remix automatically in the container, so that the adhesive remains useable, i.e. does not dry out, under the effect of the air re-entering the opened container.

1.20 Ointment- or paste-like, soft-spreading adhesives based on natural materials, more especially dextrin-based and starch-based adhesives, have been used for decades in the bonding of paper and cardboard. However, their practical application is attended by many difficulties. Their  
25 adhesive strength is limited and application of the ointment- or paste-like adhesive requires both hands because the adhesive has to be removed from its pack using a suitable tool and applied to the workpiece to be bonded. These

1 Ointment- or paste-like adhesives are normally stored in  
metal or plastics containers with a removal cover. Accord-  
ingly, the ointment or paste-like adhesive dries out undesir-  
ably quickly in the storage container.

5 For the reasons explained above, these ointment-like  
adhesives have not been as successful in practice as they  
should have been on the basis of their generally favorable  
properties. The cream-like consistency of the adhesive  
provides for optimal adaptation of the moist adhesive to the  
10 substrate to be wetted without any need for an excess of  
liquid phase which could result in undesirable saturation of  
the coated material. The soft-spreading quality of these  
ointment-like adhesives enables them to be used even in cases  
where they are difficult to apply because of restricted space.  
15 With adhesives of this type based on aqueous emulsions or  
dispersions, it is possible to vary and control the adhesive  
properties through the choice of the disperse polymer. Fin-  
ally, however, ointment- or paste-like adhesives of the  
type in question are also distinguished by the fact that they  
20 have no tendency towards legging. Any accidentally smudged  
or incorrectly applied parts of the paste-like adhesive may  
readily be wiped off before drying without leaving any remains  
of the adhesive as an obstacle to the progress of work.

The object of the present invention is to provide soft-  
25 spreading adhesives of the type in question in the form of a  
handy, ready-to-use adhesive pack which may be used in  
particular as a one-handed pack. More particularly, the  
invention seeks to utilize the advantages of adhesives based  
on emulsion or suspension polymers without being confined to  
30 the physical state of free-flowing compositions. Further  
requirements to be satisfied include substantially indefinite  
storability of the adhesive pack, easy opening and closing of  
the container for removal and after removal of the adhesive,  
easy removal and application of the adhesive in measured  
35 quantities and also free adjustability of the adhesive effect.

1 required in any particular case by optimal freedom in the  
special formulation of the particular ointment- or paste-  
like adhesive compositions used.

5 According to the invention, this object is achieved by  
the following features in combination:

- 10 a) an adhesive based on emulsion polymers thickened to an  
ointment- or paste-like consistency which, although  
soft-spreading, will not flow in the absence of pressure  
and which contain an adhesive-type homo- and/or copolymer  
stabilized with protective colloids or thickeners as  
disperse solid in aqueous phase,
- 15 b) a hand-held container accommodating the adhesive with  
an outlet opening for the adhesive and a device for the  
measured release thereof,
- 20 c) a device which reduces the adhesive-filled interior of  
the container in volume commensurate with the release  
of the adhesive in such a way that the adhesive remaining  
in the container is always bubble-free and safe against  
the re-entry of air.

In one preferred embodiment, the invention is characterized  
by the following features in combination:

- 25 a) an adhesive based on emulsion polymers thickened to an  
ointment- or paste-like consistency which, although  
soft-spreading, will not flow in the absence of pressure  
and which contain an adhesive-type homo- and/or copolymer  
stabilized with thickening protective colloids as  
disperse solid in aqueous phase,
- 30 b) a handle accommodating the adhesive and designed to be  
held in one hand,
- c) a dispenser operable by the holding hand at the head of  
the handle with an outlet opening for the adhesive,
- 35 d) a distributing element for spreading the adhesive

- 1 leaving the outlet opening onto a surface to be bonded,  
e) a device which automatically reduces the space inside  
the handle for accommodating the adhesive commensurate  
with the release of the adhesive in such a way that the  
5 adhesive remains permanently bubble-free in the handle.

The adhesives are stabilized, aqueous emulsion polymers of adhesive-type homo- and/or copolymers which are adjusted through the choice of the solids content and/or through a 10 suitable addition of thickeners to such high viscosities that the mixture of components is in the desired ointment- or paste-like, but soft-spreading form. Mixtures based on aqueous emulsion polymers suitable for the purposes of the invention have viscosities of at least about 15,000 mPas 15 (Brookfield RTV, 20°C/20 r.p.m.). More especially, the emulsion polymers in question have Brookfield viscosities of at least 50,000 mPas, the particularly preferred mixtures having viscosities of at least about 60,000 to 100,000 mPas as the lower limit of the viscosity range. The upper limit 20 of the viscosity range of the adhesives used in accordance with the invention is at about 3,000,000 mPas, as determined under comparable conditions, and preferably below 1,000,000 mPas. A viscosity range of from about 50,000 to 500,000 mPas may be particularly suitable, particularly suitable 25 mixtures frequently having viscosities in the range of from about 80,000 to 200,000 mPas (as determined at 20°C/20 r.p.m.).

The adhesives according to the invention are thus clearly distinguished from the free-flowing dispersions or emulsions of adhesive-type polymers hitherto used in the adhesives field. Free-flowing emulsion-based adhesives of the type hitherto used in practice have maximum viscosities of, for example, from 10,000 to 12,000 mPas, as determined under the conditions defined above.

Accordingly, the essence of the invention lies in the 35 novel combination of specifically selected emulsion polymers

1 of the above-mentioned type adjusted to an ointment- or paste-  
like, non-free-flowing consistency with a container which  
enables the adhesive to be dispensed in measured quantities,  
but which at the same time is designed in such a way that  
5 its adhesive-filled interior decreases in volume commensurate  
with the release of the adhesive, thus preventing air from  
re-entering or reaching the interior of the container. In  
the preferred embodiment of the invention mentioned above,  
the novel combination comprises specifically selected  
10 emulsion polymers adjusted to an ointment- or paste-like,  
non-free-flowing consistency in a dispenser held in and  
operated by one hand which acts both as a container and  
applicator for the ointment-like adhesive.

15 The use of paste-like compositions in packs of the  
type described above and more especially in dispensers held  
in and operated by one hand is known in a number of technical  
fields, but not in the field of the ointment- or paste-like,  
non-free-flowing adhesives based on aqueous emulsion polymers  
according to the present invention. The teaching of the  
20 invention is based on the observation that, under certain  
conditions, the stability in storage and indefinite reuse-  
ability of the adhesive pack are guaranteed for periods  
sufficiently long for practical requirements, even where the  
25 non-free-flowing, ointment- or paste-like emulsion polymers  
are used. This had not been expected. Adhesives based on  
emulsion polymers are not comparable in their properties of  
relevance here with other paste-like compositions, for example  
from the cosmetics field.

30 Ointment-like emulsion-based adhesives of the type in  
question tend rapidly to form a surface polymer skin in the  
presence of air. This dried polymer is ultimately the  
desired adhesive. Whereas, in known packs for adhesives  
based on free-flowing aqueous emulsion polymers, troublesome  
35 effects can be expected to be prevented or at least reduced  
through re-mixing of the entire contents of the container.

1 with any dried fractions, there is no question of any such  
regeneration by remixing in the case of ointment- or paste-  
like, non-free-flowing compositions of the type used in  
accordance with the invention. In fact, the adhesive pack  
5 had been expected rapidly to become unuseable through the  
sticking together of important parts. Surprisingly, the  
adhesive pack remains fully useable for periods of sufficient  
length for practical requirements providing the conditions  
according to the invention are observed.

10 Another problem affecting aqueous emulsion polymers of  
the type in question here lies in their possible ageing and  
increase in viscosity, even in the complete absence of air.  
Whereas this problem does not affect comparatively thinly  
15 liquid, free-flowing emulsion polymers to any significant  
extent, if at all, its occurrence in ointment- or paste-like  
compositions can result in the adhesive pack rapidly  
becoming unuseable. Problems of this nature can be avoided  
by selecting compositions of predetermined rheology, as  
reflected in the cited viscosity figures, and in particular  
20 by the choice of selected thickeners as described hereinafter.

25 The ointment- or paste-like adhesives used in accordance  
with the invention are described first in the following. In  
the preferred embodiment, the adhesive based on aqueous  
emulsion polymers has the following basic composition:

polymer solids approx. 20 - 70% by weight  
thickener or protective colloid 0.5 - 15% by weight  
emulsifiers 0 - 5% by weight  
remainder water, the water content normally being  
30 from 30 to 75% by weight.

In addition to the components mentioned above, the  
adhesives may contain other auxiliaries. Suitable  
auxiliaries are, for example, thickening agents, water-  
35 miscible substantially involatile solvents and humectants,

1 preservatives, plasticizers, pH-regulators, defoaming agents,  
film-forming aids and the like.

In the preferred embodiments, the content of adhesive-type polymer solids in the ointment-like adhesive amounts to  
5 between 35 and 60% by weight and more especially to between  
40 and 60% by weight. The preferred thickener content is normally in the range from 2 to 12% and, more especially, may amount to between 3 and 10% by weight. The use of emulsifiers within the quantitative range indicated is  
10 preferred. Humectants and/or water-soluble, substantially involatile solvents, preservatives and the like may be used in limited quantities, the upper limit for the particular auxiliary normally being about 15 to 20% by weight. However, these additional auxiliaries together preferably make up no  
15 more than 20 to 25% by weight of the mixture as a whole.  
Particular significance may be attributed to the use of limited quantities of humectants in the form of water-miscible, solid or even liquid organic components. These components may be present in quantities of, in particular,  
20 from 0.5 to 10% by weight, based on the mixture as a whole.

Suitable adhesive-type polymers include any corresponding homo- and/or copolymers which may be produced by emulsion polymerization, more especially in aqueous phase. There are many known types of polymer components such as these, of  
25 which the use is thus included within the scope of the invention. Particular significance is attributed to homo- and/or copolymers of vinyl esters, acrylic acid and/or methacrylic acid and derivatives thereof, more especially their esters, and corresponding homo- and/or copolymers of  
30 dicolefins, such as chloroprene, butadiene, isoprene and the like. However, selected styrene homo- and/or copolymers are also suitable.

Particularly important adhesives for glueing cellulose-based materials, more especially paper, cardboard and/or wood, are those based on homo- and/or copolymers of vinyl

1    esters, more especially vinylacetate. Suitable copolymers  
of vinylacetate are, for example, one or more comonomers from  
the group comprising  $\alpha$ -olefins, more especially ethylene,  
maleic acid or maleic acid derivatives, such as the  
5    corresponding anhydride, semiesters or esters of fumaric or maleic  
acid, crotonic acid, acrylic acid or methacrylic acid or  
their salts, esters or nitriles, vinylalkyl ethers of which  
the alkyl group contains from 1 to 12 and preferably from  
1 to 4 carbon atoms, styrene or N-vinyl pyrrolidone.

10    Particularly suitable vinylester copolymers contain  
the vinylester component, more especially vinylacetate,  
in quantities of at least about 40 mole % and preferably in  
quantities of at least about 50 mole %.

15    Another group of adhesive-type polymers particularly  
suitable for the purposes of the invention includes homo-  
and/or copolymers of acrylic acid and/or methacrylic acid  
or derivatives thereof, more especially their esters, amides  
and/or nitriles. Suitable comonomers are the compounds  
mentioned above in reference to polymers based on vinylacetate.

20    Styrene and/or maleic acid or maleic acid derivatives can be  
particularly suitable. As in the previously mentioned case  
of vinylester copolymers, considerable variations in the  
adhesive behavior of the end product may be obtained by  
varying the comonomer and the functional groups present  
25    therein, if any. More specifically, the relevant knowledge  
of the prior art may be applied here.

Particularly interesting adhesive-type polymer compo-  
sitions are homo- and/or copolymers based on the above-  
mentioned diolefins, more especially chloroprene.

30    The emulsion polymerization of the above-mentioned  
monomers or monomer mixtures to form the emulsion polymer  
is carried out in known manner, cf. for example Ullmann's  
Encyclopadie der technischen Chemie 4th Edition, Vol. 19,  
pages 132 to 145.

35    Protective colloids and thickeners above all up to us. 1

1 relatively small or relatively large quantities for  
stabilizing the emulsion polymers and, more particularly,  
for thickening the adhesive compositions to the desired  
ointment-like consistency. These components may actually  
5 be present in the reaction mixture during the emulsion  
polymerization reaction, although it is also possible to  
add them, in part at least, to the reaction product obtained  
after the emulsion polymerization for further stabilization  
and/or thickening. Suitable protective colloids and  
10 thickeners may be of natural origin, although corresponding  
components based on synthetic high polymers are of  
particular significance. Suitable compounds are, for  
example, water-soluble and/or water-swellable polymer  
compounds, such as hydroxyalkyl and/or carboxyalkyl celluloses,  
15 homo- and/or copolymers of acrylamide or acrylamide  
derivatives, copolymers of methylvinylether and maleic acid  
anhydride and polyvinyl alcohol.

In one particularly preferred embodiment of the invention,  
important properties of the adhesive composition are  
20 optimized by using selected thickeners or protective colloids.  
In particular, it is possible in this way to produce  
particularly smooth- and soft-spreading polymer compositions  
which are distinguished by high stability in storage, even at  
elevated temperature. Accordingly, adhesive compositions  
25 formulated with the following factors in mind are particularly  
preferred for practical application.

In this particularly preferred embodiment, the invention  
seeks to combine optimization of spreading behavior with  
optimal stability in storage of the ointment- or paste-like  
30 adhesive, even under rigorous test conditions and with the  
favorable spreading properties intact. With the desired  
paste-like consistency in mind, this behavior profile may be  
established with particular advantage when polyethylene  
glycols having a selected, average molecular weight are used  
35 for thickening. Although these polyethylene glycols may be

1 used as sole thickener, they are always present at least in  
considerable quantities.

In this embodiment, therefore, the invention relates to  
the use of polyethylene glycols having an average molecular  
5 weight of at least 3000 as a stable, non-coagulating thick-  
ener in aqueous adhesive-type emulsion polymers adjusted to  
a non-free-flowing, paste- or ointment-like consistency.

The polyethylene glycols used preferably have an average  
molecular weight of at least about 5000 and, more preferably,  
10 of at least about 10,000. The practical upper limit to the  
average molecular weight of these polyethylene glycols is a  
value of the order of 35,000, polyethylene glycols having an  
average molecular weight of the order of 12,000 to 20,000  
being particularly suitable. However, higher molecular  
15 weights, for example up to about 50,000, are not ruled out.  
In the context of the invention, "polyethylene glycols" are  
primarily understood to be homopolymers of ethylene glycol  
or ethylene oxide, although they also include copolymers  
which, to a predominant extent of preferably no less than  
20 about 50 mole %, contain ethylene oxide residues together  
with propylene oxide residues. In that case, corresponding  
block copolymers are particularly preferred.

The ointment- or paste-like aqueous emulsion polymers  
produced using polyethylene glycols of the type defined  
25 above show an optimized balance of physical properties  
immediately after their production. The ointment-like  
compositions have a gel-like consistency with the desired  
dimensional stability in the absence of external forces.  
Nevertheless, they spread particularly uniformly and easily.  
30 One factor of particular importance is that adhesives based  
on the thickeners defined herein are distinguished by very  
high stability in storage, even under difficult conditions.  
For example, the storage of correspondingly thickened aqueous  
emulsion polymers in standard laboratory equipment for a  
35 period of at least 6 months at a temperature of 50°C.

1 visible change in the physical condition of the adhesive gel. This result may be regarded as surprising. Standard commercial dispensers of the type currently on the market, for example for toothpastes, are by no means tightly sealed  
5 packs. Not only is there the outlet opening for the contents of the pack, the base which automatically moves up as the contents of the pack are squeezed out also leads to the formation of a microfissure in the wall of the pack. It could not be foreseen that storage for 6 months and longer  
10 under the difficult temperature conditions would have no effect on the physical condition of the ointment-like adhesive composition packed in the container. Under storage conditions as extreme as these, other thickeners, for example  
15 those based on cellulose derivatives or even polyvinyl alcohol and polyvinyl alcohol derivatives, actually show effects which generally culminate in more or less serious coagulation of the contents of the pack. In extreme cases, this coagulation may be so advanced as to prevent spreading of the expressed adhesive or even its portioned release.  
20 The aqueous emulsion polymers thickened using the polyethylene glycols according to the invention do not show these undesirable phenomena.

According to the invention, the polyethylene glycols are preferably present in the aqueous emulsion polymers in  
25 quantities of at least about 2% by weight and more preferably in quantities of from about 3 to 10% by weight, based on the total weight of the emulsion. As already mentioned, they may be used on their own, although they may also be mixed with other standard protective colloids with  
30 or without a thickening effect.

It can be of particular advantage to use the polyethylene glycol in combination with polyvinyl alcohol. It is possible in this way to increase the adhesive power of the adhesives, particularly when polyvinylacetate and/or a copolymer thereof is present as the adhesive-type polymer. In this

1 particular embodiment, however, it is preferred to use at  
least substantially equivalent quantities of polyethylene  
glycol and polyvinyl alcohol, although the polyethylene  
glycol is best used in excess. In this embodiment,  
5 quantitative ratios of polyethylene glycol to polyvinyl  
alcohol of from about 1 to 2 parts by weight of polyethylene  
glycol to 1 part by weight of polyvinyl alcohol for example  
give suitable protective colloids or thickeners. A  
particularly suitable polyvinyl alcohol is one having a  
10 viscosity of from about 20,000 to 60,000 mPas, as determined  
on a 4% by weight aqueous solution at 20°C.

In addition to the protective colloids, unlimited  
quantities of emulsifiers are generally used in the production  
and/or stabilization of the emulsion polymers. Thus, the  
15 polyethylene glycol-based thickeners are normally present  
together with small quantities of emulsifiers of from 0 to  
1% by weight.

Particularly suitable polymerization emulsifiers are  
anionic or nonionic types or mixtures thereof. For example,  
20 it is possible to use sulfates or sulfonates of long-chain  
alcohols or alkylphenols and also alkylbenzene sulfonates or  
sulfosuccinates. Other suitable polymerization emulsifiers  
of the type in question are sulfates of the reaction products  
of ethylene oxide with (fatty)alcohols or alkylphenols, the  
25 parent substances preferably being nonionic emulsifiers.  
Other nonionic emulsifiers are sorbitan esters of long-chain  
fatty acids, ethoxylated sorbitan esters of long-chain  
fatty acids and also alkyl glycerides.

The following further observations apply to the emulsi-  
30 fiers optionally used: whereas the protective colloids are  
generally distinguished by their high molecular weight  
structure, the emulsifiers are comparatively low molecular  
weight compounds. Suitable synthetic emulsifiers are, for  
example, anion active compounds, such as salts of relatively  
35 high molecular weight fatty acids (e.g., lauric salts of

1      sulfuric acid esters, for example sodium lauryl sulfate,  
or sulfates of alkoxylated fatty alcohols or alkylphenols,  
salts of sulfonic acids, alkylbenzene sulfonates and the  
like and also fatty adducts of various hydrophilic groups,  
5      for example protein condensates or phosphates. However,  
suitable emulsifiers may also be found among nonionic  
compounds. Suitable nonionic emulsifiers are, for example,  
partial esters of polyalcohols, for example glycerol mono-  
and distearates, sorbitol monostearate and the like,  
10     ethylene oxide or propylene oxide adducts, for example with  
fatty acids, fatty alcohols, fatty amines, partial fatty  
acid esters of polyhydric alcohols, alkylphenols and the  
like, amides of higher fatty acids, lecithins and/or steroid  
compounds, such as cholesterol and related compounds.

15     In addition to the components mentioned thus far, the  
adhesives adjusted to ointment- or paste-like consistency  
may contain further auxiliaries, for example in the form of  
thixotropizing agents. Suitable thixotropizing agents are  
insoluble inorganic compounds, for example bentonite,  
20     aluminium hydroxide, silica gels, kaolin and the like. In  
general, however, the adhesive power is affected by the  
presence of auxiliaries such as these, a factor which must  
be taken into consideration in the particular quantity in  
which these auxiliaries are used. One example of a suitable  
25     preservative is formaldehyde. Substantially involatile,  
water-miscible solvents and/or humectants are, for example,  
glycols, glycerol, partial ethers and/or esters of polyalcohols  
and the like. Suitable plasticizers, which may also be used  
for modifying the adhesive composition, are for example  
30     phthalic acid esters, acyclic dicarboxylic acid esters,  
polyesters, for example of aliphatic or aromatic dicarboxylic  
acids with diols, phosphates, such as tricresyl phosphate,  
fatty acid esters, citric acid esters or epoxide plasticizers.

35     The above-mentioned modifiers for the emulsion-based  
adhesives may be present in the reaction mixture during the

1 actual emulsion polymerization providing they do not interfere with the reaction in any way. If they are likely to have an adverse effect, they may be added to the reaction product after production of the emulsion polymer.

5 The emulsion polymer adhesives according to the invention show virtually unlimited stability in storage, i.e. they do not show any tendency towards creaming or towards phase separation under normal storage conditions, show adequate initial tack and form an adhesive bond to the required 10 extent on removal of the liquid phase in the coated material and/or when otherwise dried. In this connection, there are two basic types of adhesive effect which may be used in conjunction with one another in the adhesive. Included 15 within the scope of the invention are emulsion polymers with adhesive resins which, on setting, form strong, unbreakable adhesive bonds. However, it is also possible to introduce permanently tacky, pressure-sensitive adhesive resins into the adhesive mixtures according to the invention by emulsion polymerization. Pressure-sensitive adhesive resins of this 20 type may be present as sole adhesive resins. So far as the result of the bonding process is concerned, this implies the removability of the material attached by bonding, after which the material carrying the adhesive resin may, if desired, be subjected to another bonding process at the same 25 place or elsewhere. However, hardening adhesive resins and pressure-sensitive, permanently tacky adhesive resins may also be used in conjunction with one another. In this way, it is possible for example to vary the initial tack of the adhesive mixture and/or the open time for the bonding 30 process. Suitable permanently tacky pressure-sensitive resins are, for example, acrylate and methacrylate resins.

As mentioned earlier on, the choice of suitable containers for the adhesive compositions is an important element of the combination according to the invention. Preferred embodiments 35 of this aspect of the new adhesive pack are described in the

1 following.

A first particularly favorable and simple embodiment is characterized in that the handle is in the form of a cylindrical plastic sleeve, in the lower end of which a piston-like closure element is slidably mounted in such a way that, under the effect of the vacuum generated inside the cylindrical plastic sleeve when the dispenser is operated, the piston-like closure element automatically moves towards the dispenser at the head of the cylindrical plastic sleeve, thus ensuring that the adhesive accommodated in the handle remains bubble-free.

The outlet opening may be in the form of a permanent opening designed to be closed by means of a closure cap which also surrounds the dispenser so that the adhesive left in the inlet opening after use is prevented from drying out.

15 The outlet opening is best made of a plastics material compressible by finger pressure in order to destroy any thin film which may have formed on the surface of the adhesive in the outlet opening before the pack is re-used.

However, the outlet opening may also be in the form of a slot which is designed to open only under the pressure of the adhesive dispensed on operation of the dispenser. The mutually opposite surfaces forming the slot-like outlet opening may be kept closed under the natural tension of their constituent material or by spring means.

25 It is of particular advantage for the distributing element to surround the outlet opening on all sides and to have a substantially flat spreading surface so that, when the adhesive is being continuously dispensed through the outlet opening, the adhesive may be uniformly spread over large areas both continuously and very easily.

Two distributing channels for the adhesive tapering towards the edges of the distributing surface and intersecting at a right angle may be provided in the distributing element, the point at which they intersect coinciding with the outlet opening arranged in the middle of the distributing element. In this way, adhesive issuing continuously from the outlet opening may be uniformly applied to the surface to be treated.

1 over the entire extent of the distributing element, irrespective of the  
particular spreading direction. However, this does presuppose that, after  
each use, the spreading surface is wiped briefly with the tapering dis-  
tributing channels to prevent adhesive hardening in the channels which should  
5 best be done in any case when the spreading surface is not provided with  
prescribed distributing channels to promote clean spreading during the next use.

In one particularly simple embodiment, the dispenser comprises an elastically compressible and, after release, returning element at the head of the cylindrical plastic tube and the piston-like closure element is prevented from moving in the direction which would enlarge the interior of the cylindrical plastic sleeve. The dispenser does not require any further means when, as described above, the outlet opening is in the form of a slot which only opens under the pressure of the adhesive dispensed on operation of the dispenser.

However, if the outlet opening is in the form of a permanent opening, the dispenser - in addition to an elastically compressible and, after release, returning element at the head of the cylindrical plastic sleeve - advantageously comprises a non-return valve between the compressible element and the permanently open outlet opening, the piston-like closure element again being prevented from moving in the direction which enlarges the interior of the cylindrical plastic sleeve.

The non-return valve preferably has a valve seat against which a flap hinged on one side sealingly bears under natural tension.

In another embodiment, the dispenser may comprise a compressible and, after release, returning element and a pumping chamber arranged between that element and the remaining interior of the plastic sleeve, a non-return valve which closes on compression of the element and opens on the return thereof being arranged between the pumping chamber and the rest of the interior and another non-return valve which opens on compression of the element and remains fully

1 adhesive and which closes on the return of the element being  
arranged between the pumping chamber and the outlet opening.  
This embodiment eliminates the need for a separate mechanism  
to lock the piston-like closure element on operation of the  
5 dispenser.

Finally, in another embodiment, the dispenser may be provided at the head of the plastic sleeve with a hollow mouthpiece which includes the outlet opening and which is designed to be compressed together with an elastic element of 10 the plastic sleeve, that end of the hollow mouthpiece facing the interior of the plastic sleeve being closed apart from valve openings which, in the returned position of the element, are closed by surfaces slidably guiding the mouthpiece and which communicate with the interior of the plastic 15 sleeve on compression of the mouthpiece together with element, the piston-like closure element being prevented from moving in the direction which enlarges the interior of the plastic sleeve.

The invention is described in more detail in the following 20 with reference to the accompanying drawings, wherein:

Figure 1 is a longitudinal section through one embodiment of an adhesive pack according to the invention in which the outlet opening is closed before operation of the dispenser.

Figure 2 is a partial section corresponding to Figure 1 with the outlet opening in its open position after operation 25 of the dispenser.

Figure 3 is a longitudinal section through the head region of another embodiment of an adhesive pack according to the invention.

Figure 4 is a longitudinal section through another embodiment of an adhesive pack according to the invention.

Figure 5 is a section on the line A-A of Figure 4 in the inoperative position of a dispenser provided there.

Figure 6 is a section corresponding to Figure 5 in the 30 working position of the dispenser.

1       Figure 7 is a partial longitudinal section through an  
embodiment modified somewhat in relation to the embodiment  
shown in Figures 4 to 6.

5       Figure 8 is an elevation in the direction of arrow B in  
Figure 7.

Figure 9 is a partial longitudinal section through  
another embodiment of an adhesive pack according to the  
invention in the inoperative position of a dispenser  
provided there.

11      Figure 10 is a partial longitudinal section similar to  
Figure 9 in the working position of the dispenser.

In the embodiment shown in Figures 1 and 2, a piston-like closure element 2 is provided near one end of a cylindrical sleeve 1, being provided with a spreading spring 3 of which the ends 4 bear resiliently against the inner wall of the cylindrical sleeve 1 in such a way that the piston-like closure element is able to move to the left in Figure 1, but is prevented from moving to the right in Figure 1. Arranged at the other end, i.e. at the head, of the cylindrical sleeve 1 is an elastically depressible element 5 which is kept in the starting position shown in Figure 1 by means of a return spring 6. In the element 5, there is a slot-like outlet opening 7 which is kept in the closed position shown in Figure 1 by a U-shaped spring 8. The slot-like opening 7 is surrounded on all sides by a distributing element 9 which has a substantially flat spreading surface. The interior of the cylindrical sleeve 1 and of the depressible element 5 is filled with an ointment- or paste-like adhesive of the type described above. The adhesive pack is operated very easily by taking up the cylindrical sleeve 1 serving as a handle in one hand and depressing the compressible element 5, preferably using the thumb of the holding hand, against the action of the return spring 6, the slot-like opening 7 opening under the effect of the pressure generated in the interior and releasing

1 adhesive, as shown in Figure 2, whilst the piston-like closure element 2  
is prevented by spreading spring ends 4 from moving to the right in Figure 1  
under the internal pressure in question. After the compressible element 5  
has been released, it returns under the action of the return spring 6 from  
5 the position shown in Figure 2 to the starting position shown in Figure 1,  
the slot like opening 7 being the first to close and, under the effect  
of the resulting increase in the volume of the interior of the pack, a  
reduced pressure builds up therein, causing the piston like closure element 2  
to move to the left in Figure 1 by a distance corresponding to the amount  
10 of adhesive dispensed. As can be seen, the dispensing operation in question  
may be repeated any number of times and the adhesive issuing through the  
outlet opening 7 uniformly distributed by means of the distributing element 9  
over a surface to be bonded.

Figure 3 shows another embodiment of the head section of an adhesive  
15 pack in the form of only one elastically compressible and, after release,  
returning element 10 with which a cylindrical sleeve (not shown) having a  
piston-like closure element is designed to communicate in the same way  
as in the previously described adhesive pack shown in Figures  
1 and 2. Integrally formed on the compressible element 10  
20 is a mouthpiece 11 at the end of which there is a  
permanently open outlet opening 12 formed by surrounding  
plastics material compressible by finger pressure, the  
surrounding ring-shaped end faces 13 forming a distributing  
element by means of which adhesive issuing from the opening  
25 12 can be uniformly distributed over a surface to be bonded.  
Inside the mouthpiece 11 there is a valve seat 14 against  
which a valve flap 15 pivotally connected on one side to the  
mouthpiece sealingly bears under natural tension. The  
mouthpiece is provided on top with a knurled pressing surface 16.  
To operate the described adhesive pack, the flexible  
element 10 is depressed so that the valve 15 opens  
under the pressure generated and adhesive is dispensed from  
the outlet opening 12, whereas when the pressing surface 16 is  
released the valve 15 initially closes under its natural  
35 tension so that a reduced pressure is built up inside the  
adhesive pack, causing the piston-like closure element

1 arranged at the other end of the adhesive pack to follow up  
in the manner previously described in reference to the  
embodiment illustrated in Figures 1 and 2.

In the embodiment of an adhesive pack shown in Figures  
5 4 to 6, a cylindrical plastic sleeve 17 is again provided  
at its lower end with a piston-like closure element 18 and,  
at its upper end, with a headpiece 19 in which a piston-like,  
depressible element 20 is slidably mounted. When it is  
depressed, the element 20 reduces the volume of a pumping  
10 chamber 21 formed in the headpiece 19, a U-shaped return  
spring 22 moving the piston-like element 20 back upwards  
after its release, as shown in the drawing. In addition,  
an outlet passage 23 is formed in the headpiece 19,  
terminating in an outlet opening 24 designed to be closed by  
15 a screw cap 25. The outlet passage 23 communicates with  
the pumping chamber 21 through an opening 26. This opening 26  
is formed in an injection-molded part 27 which is integrally  
formed with a valve flap 28 hinged on one side and with another  
valve flap 29, the valve flap 29 being used to block the  
outlet passage 23 and the valve flap 28 being used to block  
and release an opening 30 between the interior of the  
cylindrical sleeve 17 and the pumping chamber 21. To operate  
the adhesive pack described in the foregoing, the piston-like  
element 20 is depressed from the position shown in Figs. 4  
25 and 5 into the position shown in Fig. 6 so that, with the  
opening 30 blocked by the valve flap 28 and the valve flap 29  
opening in the outlet passage 23, adhesive situated in the  
pumping chamber 21 and in the outlet passage 23 is forced out  
through the outlet opening 24. When the piston-like element  
30 20 is released, the volume of the pumping chamber 21 is  
increased and a reduced pressure is built up therein, opening  
the valve flap 28 and closing the valve flap 29 so that,  
as the piston-like closure element 18 moves up, adhesive  
passes from the interior of the cylindrical sleeve 17 into  
35 the pumping chamber 21 in which it waits to be dispensed.

1 next time the piston-like element 20 is depressed. As can  
be seen, there is no need in this embodiment for the piston-  
like closure element 18 to be provided with means which prevent  
it from moving downwards in Fig. 4 because the valve flap 28  
5 screens off the interior of the tube 17 against the pressure  
building up in the pumping chamber 21 when the piston-like  
element 20 is depressed. Each time a certain amount of  
adhesive issues from the outlet opening 24, it may be  
uniformly distributed over a surface to be bonded by means  
10 of a distributing element 31 formed laterally on the head-  
piece 19.

In order further to simplify this distributing-spreading process, the embodiment shown in Figs. 7 and 8, which is only slightly modified in relation to the embodiment shown 15 in Figs. 4 to 6, comprises an outlet opening 32 formed laterally in the headpiece 19a and, to be more precise, centrally in a circular distributing element 31a, the construction otherwise corresponding to that illustrated in Figs. 4 to 6. As can be seen, it is possible by virtue 20 of this construction for adhesive to be continuously dispensed through the outlet opening 32 by corresponding repeated depression of the piston-like element 20 and, at the same time, uniformly applied to and spread over a surface to be bonded.

Finally, Figs. 9 and 10 show another embodiment in 25 which only the upper region of the adhesive pack in question is illustrated. Figure 9 shows the pack before operation whilst Fig. 10 shows the pack in its operated position.

Once again, there is a cylindrical plastic sleeve 33 in the 30 lower end of which is arranged a piston-like closure element (not shown) which, as in the embodiments shown in Figs. 1 to 3, is prevented from moving in the direction which would increase the interior of the sleeve in volume. At the head of the cylindrical sleeve 33, there is again an elastically deformable element 34 to which an annular hollow mouthpiece 35 is connected. In the annular part, the mouthpiece 35

1 provided with an outlet opening 36 designed to be closed  
2 by a closure cap 37 when the pack is not in use. The other  
3 angled end of the mouthpiece 35 is continuous apart from  
4 valve openings 38, these valve openings 38 being covered or  
5 closed by an annular guide element 39 in the inoperative  
6 position shown in Fig. 9. The mouthpiece 35 is further  
7 provided with an upper pushbutton 40. To operate the  
8 adhesive pack in question, the mouthpiece 35 is depressed  
9 by means of the pushbutton 40 from the position shown in  
10 Fig. 9 into the position shown in Fig. 10 so that the  
11 valve openings 38 in the mouthpiece are released and  
12 communicate with the interior of the adhesive pack, enabling  
13 adhesive to pass from that interior through the valve  
14 openings 38 into the interior of the mouthpiece 35 from  
15 which it is forced out through the outlet opening 36 under  
16 the effect of the pressure building up when the mouthpiece 35  
17 is depressed, the piston-like closure element at the other  
18 end of the cylindrical sleeve 33 being prevented from moving  
19 outwards in the manner already described. When the push-  
20 button 40 is released, the valve openings 38 are again  
21 closed by the guide ring 39 and, under the effect of the  
22 vacuum building up inside the pack during the return move-  
23 ment of the flexible element 34, the piston-like closure  
24 element (not shown) at the lower end of the cylindrical  
25 sleeve 33 moves up in accordance with the amount of adhesive  
26 dispensed. This operation may also be repeated any number  
27 of times as required according to the desired application  
28 of adhesive, the adhesive dispensed being distributed onto  
29 the surface to be bonded either by the end faces of the  
30 mouthpiece 35 surrounding the outlet opening 36 or by  
31 a distributing element (not shown) - similar to the  
32 distributing element 31 in the embodiment shown in Figs. 4  
33 to 6 - formed at a suitable place on the head of the  
34 adhesive pack.

35 However, the non-free-flowing adhesive polymers indicated

1 to the consistency of an ointment or paste may also be used  
in other containers, i.e. in dispensers or containers without  
a metering facility at the head of the container, without  
the stability of the particular adhesive in storage being  
5 restricted unacceptably for practical requirements and without  
the practicability of the adhesive pack being seriously  
impaired.

Above all, it has been found that the stability in storage  
of the non-free-flowing emulsion polymers adjusted to the  
10 consistency of an ointment or paste is not impaired over  
periods of sufficient length for practical requirements  
providing the adhesive remaining in the pack is always safe  
against the re-entry of air into the pack.

A distributing element for spreading the adhesive  
15 leaving the outlet opening onto a surface to be bonded  
may advantageously be provided on the container as in the  
previously described embodiment. However, this is not  
absolutely essential because, depending on the type of  
dispenser used, for example a narrow dispensing nozzle, the  
20 adhesive in question may also be applied in difficult corners  
and the like, i.e. without requiring subsequent spreading.

In a particularly simple, but very easy-to-handle  
embodiment, the container is in the form of an elastically  
deformable tube between which and the adhesive is arranged a bag-  
25 like, air-tight, permanently or inelastically deformable  
film and at least one vent opening is provided in the  
elastically deformable tube.

It has been found that, in this embodiment, it is possible  
to use simply and inexpensively manufactured plastic tubes  
30 of the type already used, for example, for hair-washing  
preparations and other light-duty detergents where it does  
not matter if, after the required amount of preparation has  
been squeezed out, air enters the tube and mixes into contact  
with the remaining contents so that when the tube expands  
35 back into shape. In the preferred embodiment, the tube

1 air is prevented from being sucked in thus by the internal  
5 bag-like film because it retains the compressed configuration  
it assumed on compression of the outer elastic tube.

10 It has proved to be of advantage for the permanently or  
15 inelastically deformable film to be not only impermeable to  
air but also impermeable to water vapor. Particularly  
suitable films which meet this requirement are films of  
low-density, medium-density and high-density polyethylene  
and oriented and non-oriented polypropylene.

20 In one particularly practical and simple embodiment,  
the film is a laminate of which one part is a plastics  
film weldable to an elastically deformable plastics tube  
whilst the other part is a permanently and inelastically de-  
formable film impermeable to air and water vapor. The  
25 weldable plastics film may consist, for example, of poly-  
ethylene or non-oriented polypropylene film.

30 However, polyester films impermeable to water vapor,  
for example polyethylene glycol terephthalate films, may  
also be used. In addition, films of any other type  
35 (polyesters, polyamides, Cellophane) may be made impermeable  
to water vapor by coating with polyvinylidene chloride  
(on one or both sides).

40 If the outer elastic tube is made of material which is  
not intended to be heat-shrinkable or weldable with the internal  
plastics films, the tube material may even be coated with  
45 a hotmelt adhesive in the region where it is joined to the  
film, i.e. in the vicinity of the mouthpiece, in order to  
enable the tube and the film to be joined to one another  
simply by applying heat.

50 Another embodiment of the invention is characterized in  
that the container is in the form of a cylindrical plastic  
55 sleeve in that end of which facing the outlet opening is  
arranged a piston which is rotatable to be rotated from  
the outlet opening. It is possible to provide the housing  
of the piston with a number of longitudinal slits.

1        Finally, in another embodiment, the piston may be  
driven by a bellows pump which feeds compressed air to a  
compression chamber formed on that side of the piston oppo-  
site the adhesive.

5        A nonreturn valve may advantageously be arranged  
between the bellows pump and the compression chamber.

In addition, the bellows pump may be laterally  
arranged on the outside of the cylindrical plastic sleeve  
in such a way that it may be operated by the finger of  
10      the hand holding the plastic sleeve, making the adhesive  
pack particularly convenient to handle.

When adhesive packs of the type in question are stored  
under extreme conditions, particularly over very long periods  
and/or at high temperatures and/or under very dry conditions,  
15      it may be desirable to counteract a possible loss of water  
from the emulsion polymers by completely or substantially  
completely preventing the diffusion of water vapor through  
the walls of the solid container material. To this end,  
20      the container may be made of plastics impermeable to the  
diffusion of water vapor or metal containers may be used.  
25      Basically, it is known that solid plastics can differ in  
their impermeability to water vapor. Thermoplastic plastics  
highly impermeable to the diffusion of water vapor are known  
(cf. Polymer Handbook, 2nd Ed. (1975), John Wiley & Son, N.Y.).  
Accordingly, high-density polyethylene, polyvinylidene  
chloride, polypropylene, polyisoprene and others are  
particularly suitable.

Where metal containers are used, they may be made of  
a variety of metals, including in particular lightweight  
30      metal alloys. For example, cylindrical aluminum tubes  
may be used. Other ductile metals, such as copper or zinc  
and alloys thereof, may also be used for special problems.

Depending on the requirements it is satisfied, it may  
be appropriate to use the materials mentioned above in  
35      combination with one or more of the following, i.e.,

a particularly smooth material merging tightly with the bottom plate may be used for the interior of the container whilst a stabilizing material which lends itself to printing or adhesive labelling is used for the outside. Also, the container accommodating the adhesive need not necessarily be tubular in shape. Other cross-sections, such as for example oval or hexagonal, could also be considered.

In addition, the container need not be made entirely of materials correspondingly impermeable to water vapor. Design factors may make it appear desirable for individual components, for example the container head with its pump system, to be made of a material particularly suited to its construction without any regard for the water-vapor diffusion values. However, emphasis may be placed on this aspect of high impermeability to water vapor in the construction of all the components of the dispenser.

In support of these efforts to obtain high impermeability levels for the container, another embodiment of the invention is characterized in that the one-hand adhesive pack is in the form of a container with an uninterrupted base, a piston-like closure element being slidably arranged within the container at the lower end thereof. In this embodiment, the closure element may be mounted with a permanent bias in the emptying direction. This is made possible for example by a compression spring inside the container between the bottom thereof and the underneath of the piston-like closure element and/or by the provision of a pressurized propellant gas at a suitable point of the container. When the dispenser is operated by the hand holding the pack, the piston-like closure element under emptying pressure forces adhesive out of the outlet opening in the head of the pack until operation of the dispenser is interrupted again.

In this embodiment, too, the adhesive remaining in the container always adheres to the surface on which it is contained, thereby preventing it from accidentally

1 until there is no more adhesive left in the container.  
In this connection, it is of course important to ensure  
that adequate emptying pressure is maintained until the  
requisite amount of adhesive has been dispensed.

EXAMPLES

a) Production of dispersions in 30 kg batches

5       The apparatus used was a 50 liter capacity glass apparatus equipped with an infinitely variable anchor stirrer, a 20 liter metering vessel and an ascending reflux condenser. After each batch, the apparatus was rinsed with water and then acetone and dried.

10

Procedure

15       Most of the aqueous phase was introduced into the reactor and sodium bicarbonate, emulsifier, defoamer and protective colloid were dissolved therein for 2 hours at 75°C (starting solution). The monomer or mixture of monomers was introduced in the prescribed quantity into the metering vessel.

20       The initiator (potassium persulfate) was dissolved in the rest of the water and the solution introduced into another metering vessel.

25       To carry out polymerization, monomer and initiator solution were simultaneously introduced into the reactor at 82 to 88°C at the rates shown in the following Table.

Table I

Time (mins.)	Initiator solution added (l)	Monomer added (l)
0	0	0
20	0.4	1.6
30	0.4	1.6
60	0.8	4.6
120	1.2	7.6
160	1.6	10.4
240	2.0	13.6
300	2.4	13.6

1       The reaction temperature was kept in the range from  
80 to 88°C. In the event of over-vigorous refluxing, the  
input of monomer was slowed down. After the monomer had  
been added, the temperature was increased to 85-95°C for  
5      the after-reaction. The dispersions could be packed after  
cooling to approximately 35°C.

b) Production on a laboratory scale

10     The apparatus used was a 2 liter capacity face-ground  
glass apparatus equipped with a metal anchor stirrer, an  
intensive cooler and metering vessels. Most of the water  
was introduced into the reaction vessel and the sodium  
bicarbonate, the emulsifier, the defoamer and the protective  
colloid were dissolved therein for 2 hours at 80 to 85°C.  
15     The monomers were introduced into a 0.5 liter metering  
funnel. To prepare the initiator solution, potassium  
persulfate was dissolved in cold water. The solution was  
introduced into another metering funnel.

20     To carry out polymerization, monomer and initiator  
solution were simultaneously introduced into the reaction  
vessel at 82 to 88°C at the rates shown in Table 2 below:

Table 2

Time (mins.)	Initiator solution added (ml)	Monomer added (ml)
0	0	0
20	20	50
40	20	100
30	60	175
120	50	300
180	65	425
240	75	550

35     The reaction was carried out with stirring. The  
reaction temperature was kept in the range from 80 to 88°C.

1 In the event of over-vigorous refluxing, the input of  
monomer was temporarily reduced. After the monomer had been  
added, the rest of the initiator solution was quickly added  
and the end of refluxing awaited. The after reaction then  
5 took place over a period of 30 minutes at a temperature of  
from 85 to 95°C.

EXAMPLE 1

Raw material class	Raw material	Concentration used % by weight	kg
Water		41.8	12.540
Emulsifier	nonylphenol-10 EO-sulfate, 30% NaHCO <sub>3</sub>	0.3 0.2	0.300 0.060
Defoamer	commercial grade	0.05	0.015
Protective colloid	PEG 20000	7.0	2.100
Monomer	vinyacetate	42.20	12.660
Initiator solution	potassium persulfate	0.25	0.075
	water	7.5	2.250

Viscosity:  
(Brookfield RVT,  
25 20°C/20 r.p.m.)

35000 mPas

Appearance  
(film/glass):

clear, speck-free

Flexibility:

brittle-elastic

Film/water behavior:

resistant.

30 Particle size:

ca. 250 nm

Solids content:

49.4%

EXAMPLE 2

35 The procedure was as in Example 1, except that, instead of 0.3% by weight of emulsifier, only 0.1% by weight of the

1 same emulsifier was used.

5      Viscosity:                            22000 mPas  
      (Brookfield RVT  
      20°C/20 r.p.m.)

10     Appearance:                          clear, very few specks  
      (film/glass)

15     Flexibility:                        brittle-elastic

20     Film/water  
      behavior:                            resistant

25     Particle size:                    330 nm

30     Solids content:                    50.0%

EXAMPLE 3

15 Example 1 was repeated on a 1 kg laboratory scale, except  
      that, instead of 7.0% by weight of a protective colloid  
      having a molecular weight of 20,000, 10.0% by weight of a  
      protective colloid having a molecular weight of 12,000 was  
      used. The vinylacetate was used in a quantity of 39.2% by  
      weight.

20     Viscosity:                            32000 mPas  
      (Brookfield RVT,  
      20°C/20 r.p.m.)

25     Appearance:                        clear, speck-free  
      (film/glass)

30     Flexibility:                        only slightly elastic

35     Film/water  
      behavior:                            resistant

40     Particle size:                    160 nm

45     Solids content:                    ~50%

1    EXAMPLE 4

	Raw material class	Raw material	Concentration used by weight	kg
5	Water		41.34	12.402
	Emulsifier	nonylphenol-10 EO-sulfate, 30% NaHCO <sub>3</sub>	0.50 0.20	0.498 0.060
	Defoamer	commercial grade	0.05	0.015
10	Protective colloid	PEG 20000	7.0	2.100
	Monomer	vinyacetate	42.0	12.600
	Initiator solution	potassium persulfate	0.25	0.075
		water	7.5	2.250

15

	Viscosity: (Brookfield RVT, 20°C/20 r.p.m.)	140000 mPas
	Appearance: (film/glass)	clear, speck-free
20	Flexibility:	elastic
	Film/water behavior:	resistant
	Particle size:	ca. 300 nm
	Solids content:	50.1%

EXAMPLE 5

	Raw material class	Raw material	Concentration used % by weight	kg
1	Water		34.17	10.251
5	Emulsifier	nonylphenol-10 EO-sulfate, 30% NaHCO <sub>3</sub>	1.0 0.2	0.999 0.060
	Defoamer	commercial grade	0.2	0.060
	Protective colloid	PEG 12000	3.5	1.050
10	Monomer	vinylacetate dibutylmaleate	33.56 17.29	10.068 5.187
	Initiator solution	potassium persulfate	0.25	0.075
		water	7.5	2.250

15

Viscosity:  
(Brookfield RVT,  
20°C/20 r.p.m.)

19000 mPas

Appearance:  
(film/glass)

clear, speck-free

20

Flexibility:

elastic

Film/water behavior:

resistant

Particle size:

ca. 330 nm

Solids content:

55.7%

1    EXAMPLE 6

	Raw material class	Raw material	Concentration used % by weight	kg
5				
	Water		36.5	12.251
	Emulsifier	nonylphenol-10 EO-sulfate, 30%	1.0	0.999
		NaHCO <sub>3</sub>	0.19	0.057
10	Defoamer	commercial grade	0.10	0.030
	Protective colloid	PEG 12000	5.0	1.500
	Monomer	vinylacetate	32.64	9.792
		dibutylmaleate	16.81	5.043
15	Initiator solution	potassium persulfate	0.25	0.075
		water	7.5	2.250

20    Viscosity:  
(Brookfield RVT,  
20 °C/20 r.p.m.)

34000 mPas

25    Appearance:  
(film/glass)

clear, speck-free

Flexibility:

elastic

20    Film/water behavior:

resistant

25    Particle size:

ca. 300 nm

30    Solids content:

36.4%

EXAMPLES 7 to 20

30    The dispersions of Examples 7 to 20 were prepared on a 1 kg laboratory scale in the same way as in Example 3. The dispersions of Examples 7 to 14 gave brittle films whilst the dispersions of Example 15 to 20 gave elastic films. All products were virtually speck-free and gave water-resistant films. Further data are shown in Table 4.

Table 3

Polyvinylacetate dispersions (Examples 7 to 20)

	Vinylacetate % by weight	Dibutylmaleate % by weight	PEG % by weight	Nonylphenol-10 EO- sulfate, 30% % by weight	Defoamer % by weight
Example 7	44.0	-	20000	5	0.5
Example 8	39.0	-	20000	1.0	0.5
Example 9	39.5	-	20000	1.0	-
Example 10	42.2	-	20000	7	0.3
Example 11	39.0	-	12000	1.0	0.5
Example 12	39.25	-	12000	1.0	0.25
Example 13	42.2	-	20000	7	0.3
Example 14	42.0	-	20000	7	0.5
Example 15	34.26	17.68	20000	2.5	1.0
Example 16	34.55	17.80	20000	2.5	0.5
Example 17	33.56	17.29	12000	3.5	1.0
Example 18	33.20	17.16	12000	4.0	1.0
Example 19	32.64	16.81	12000	5.0	1.0
Example 20	33.96	17.50	12000	3.0	1.0

Table 3 continued

Viscosity (Brookfield RVT, 20 °C/20 r.p.m.)	Particle size nm	Solids content % by weight
Example 7	33000	350
Example 8	61000	220
Example 9	13750	200
Example 10	49000	280
Example 11	27500	184
Example 12	10000	187
Example 13	190000	-
Example 14	170000	-
Example 15	92000	620
Example 16	25000	600
Example 17	56000	180
Example 18	80000	680
Example 19	158000	360
Example 20	180000	320

1    EXAMPLE 21

	Raw material class	Raw material	Concentration used % by weight	kg
5	Water		39.15	11.061
	Emulsifier	nonylphenol-10 EO-sulfate	1.00	0.999
		NaHCO <sub>3</sub>	0.19	0.057
10	Defoamer	Nopco 8034	0.10	0.030
	Protective colloid	PEG 20000	7.00	2.100
		Mowiol 4-88 *)	5.00	1.500
	Monomer	vinylacetate	26.24	7.872
		dibutylmaleate	13.54	4.062
15	Initiator	potassium persulfate	0.25	0.075
	Water		7.5	2.250

Viscosity: 198000 mPa.s

(Brookfield RVT,

20 20°C/20 r.p.m.)

Appearance: slightly clouded, very few specks  
(film/glass)

Flexibility: elastic

Film/water behavior: resistant

25 Particle size: 270 nm

Solids content: 52.4%

\*) A polyvinyl alcohol made by Hoechst AG.

1    EXAMPLE 22

	Raw material class	Raw material	Concentration used % by weight	kg
5	Water		22.55	0.2022
	Emulsifier	nonylphenol-10 EO-sulfate	1.0	0.0333
		NaHCO <sub>3</sub>	0.2	0.0020
10	Defoamer	Nopco 8034	0.2	0.0020
	Protective colloid	Pluronic P105*)	3.5	0.0350
	Monomer	vinylacetate	42.8	0.4280
		dibutylmaleate	22.0	0.2200
15	Initiator	potassium persulfate	0.25	0.0025
	Water		7.5	0.0750

20                  Viscosity:                100000 mPa.s  
                        (Brookfield RVT,  
                        20°C/20 r.p.m.)

25                  Appearance:              clear, very few specks  
                        (film/glass)

                        flexibility:           elastic

                        Film/water behavior:     resistant

30                  Particle size:           ca. 230 nm

                        Solids content:       70.2%

\*) A polyoi made by Wyandotte Chemicals Corp. Wyardolle/  
Michigan, USA, gram-molecular weight ca. 6500 g/mole,  
polyoxyethylene content 50%

1    EXAMPLE 23

693.9 g of water were introduced into the reaction vessel according to b) and 26.6 g of nonylphenol-10 EO-sulfate, 30%, 6 g of potassium persulfate and 100 g of PEG 5 20000 were dissolved therein under nitrogen while stirring over a period of 30 minutes at 85°C.

A monomer mixture consisting of 175 g of ethylacrylate and 20 g of acrylic acid was then introduced dropwise with stirring (140 r.p.m.) over a period of 2.5 hours at 87 to 10 89°C.

Following a 30-minute after-reaction, 1.25 g of hydrogen peroxide in 1.27 g of water was added as catalyst and, after another 30 minutes' reaction at 89°C, 26 g of fatty alcohol-50 EO dissolved in 27 g of water were added and the mixture 15 stirred for 15 minutes.

The dispersion was cooled to 40°C at 140 r.p.m. and neutralized over a period of 10 minutes with 20 g of ammonia (25%).

Following another 20 minutes' after-reaction, the 20 dispersion was cooled to 20°C and packed.

1	Raw material class	Raw material	Concentration used % by weight	kg
	Water		66.76	0.6939
5	Emulsifier	nonylphenol-10		
		EO-sulfate	0.77	0.0266
		fatty alcohol-50 EO	1.25	0.0260
	Protective colloid	PEG 20000	9.62	0.100
10	Monomer	ethylacrylate	16.84	0.175
		acrylic acid	1.93	0.020
15	Initiator	potassium persulfate	0.58	0.006
		hydrogen peroxide (50%)	0.058	0.00125
		Water	0.265	0.027
15	Neutralizing agent	ammonia (25%)	1.93	0.020

20	Viscosity: (Brookfield RVT, 20°C/20 r.p.m.)	45000 mPas
	Appearance: (film/glass)	slightly clouded, speck-free
	Flexibility:	elastic
25	Film/water behavior:	resistant
	Solids content:	39.2%

1    EXAMPLE 24

281 g of water were introduced into the reaction vessel according to b) and 45 g of nonylphenol-10 EO-sulfate (30%), 0.5 g of potassium persulfate and 100 g of PEG 20000 were dissolved under nitrogen therein over a period of 30 minutes with stirring at 76°C.

A monomer mixture consisting of 218 g of ethylacrylate and 38.7 g of styrene was then introduced dropwise together with the catalyst solution, consisting of 175 g of water, 10 1 g of potassium persulfate, 43.3 g of nonylphenol-10 EO-sulfate, 30%, and 24.7 g of sodium hydroxide (20%), over a period of 2 to 2.5 hours at 87 to 90°C/140 r.p.m. The after-reaction took place over a period of 30 minutes at 87 to 90°C.

15    After cooling to 25°C, the product was packed.

1	Raw material class	Raw material	Concentration used % by weight	kg
	Water		55.35	0.4535
5	Emulsifier	nonyphenol-10 EO-sulfate, 30%	0.32	0.0883
	Protective colloid	PEG 20000	12.22	0.100
	Monomer	ethylacrylate	26.61	0.2180
		styrene	4.72	0.0387
10	Initiator	potassium persulfate	0.18	0.0015
	Neutralizing agent	sodium hydroxide, 20%	0.60	0.0247

15 Viscosity: 1 800 000 mPas  
(Brookfield RVT,  
20°C/1 r.p.m.) 48 000 mPas (diluted 1:1 with H<sub>2</sub>O)

Appearance (film/glass),  
diluted 1:1 with water: slightly clouded, slightly speckled

Flexibility: elastic

20 Film/water behavior: resistant

Solids content: 40.7%

Methods used to determine the properties of the dispersions

25 Solids content

The solids content is determined by means of a Mettler PC 440 balance in conjunction with a Mettler CP 12 solids attachment at heating stage 4 (approx. 115°C); drying time 30 mins.

30 pH-value

The pH-value is determined at 20°C using a Seibold No. 6702483 pH meter.

Viscosity (Brookfield)

35 The Brookfield viscosity is measured at 20°C in a Brookfield RVT 2-4/20/20 viscosimeter.

1 Viscosity (Epprecht)

The Epprecht viscosity is measured at 20°C in an Epprecht STV viscosimeter, spindle 2 or 3.

5 Particle size

Particle size is determined in an approx. 1000-fold dilution with 1% sodium chloride solution in a Coulter Nano-Sizer TM. The average value of 3 measurements is expressed as the particle size.

10

Appearance of the film after drying at 100°C

The dispersion is drawn with an Erichsen model 288 film drawer onto a glass plate in a layer thickness of 80 my and dried for 3 mins at 100°C. After standing for 30 mins at 15 room temperature, the appearance of the dispersion film is optically assessed.

Properties of the film after storage in water

The dispersion film having a wet layer thickness of 20 80 my, which had been previously stored for 30 mins. at 100°C in a drying cabinet and then for 30 mins. at room temperature, is placed for 10 minutes in water having a temperature of 20°C.

- a) Resistance to water is then assessed by rubbing whilst  
25 b) elasticity is assessed by drawing the wet film.

Useability of the adhesives after storage

The adhesives of Examples 1 - 24 are packed in standard commercial plastic dispensers comprising a metering device with an outlet opening for the adhesive at the head of the 30 container operable by the hand holding the adhesive pack and a base which moves up automatically as the adhesive is dispensed.

The expressability of the adhesives from the dispenser is unchanged even after storage for 180 days at room temperature. Intensified ageing at 40°C also shows that the adhesive pack remains useable over the same period.

1. EXAMPLE 25

Ointment- or paste-like adhesives based on aqueous emulsion homo- and copolymers of vinyl acetate are prepared as follows from the following prototype formulation:

5       The particular protective colloid selected (hydroxyethyl cellulose, polyvinyl alcohol, carboxymethyl cellulose or polyvinyl pyrrolidone) is stirred into cold demineralized water, heated if desired to ca. 80°C and homogenized by an adequate residence time at that temperature. An initiator 10 solution for initiating the polymerization reaction is separately prepared by dissolving potassium persulfate in demineralized water at ca. 40°C.

The aqueous solution containing the protective colloid is introduced into the reactor and stirred therein at 150 r.p.m.

15     Introduction of the monomer and introduction of the initiator are commenced at the same time at approx. 75°C. Both components are introduced over a period of about 3 hours, 10% of the initiator solution being kept back for the after-reaction. During the after-reaction, the initiator is 20 added about 10 after the last addition of monomer (after-reaction time ca. 30 minutes). Throughout the polymerization reaction, including the after-reaction, the reaction temperature should not significantly exceed 80°C.

25     The dispersions obtained as reaction product are substantially speck-free and dry to form solid films. The product viscosities (Brookfield 20°C/20 r.p.m.) extend into the 3 million range, depending on the type and concentration of the protective colloid.

30     In the following Table, adhesives 1-3 and 5 are homo-polymers of vinyl acetate and adhesives 4 and 6 copolymers of 2/3 parts by weight of vinyl acetate and 1/3 part by weight of dibutyl maleate.

1

Table

		Emulsifier	Quantity % by weight	Solids % by weight	Brookfield viscosity
5	1	Hydroxyethyl cellulose	2.5	47	65 000
	2	Hydroxyethyl cellulose	3.0	50	110 000
10	4	Polyvinyl alcohol	7.0	48	2 700 000
	5	Polyvinyl alcohol	7.0	57	3 600 000
	5	Carboxymethyl cellulose	5.0	50	31 500
	6	Carboxymethyl cellulose	4.0	51	65 800

15       The ointment-like adhesives are introduced into a container in the form of a cylindrical plastic sleeve which, at its lower end, comprises a slidably mounted piston-like closure element. The container further comprises, as a metering device, a depressible and, after release, automatically returning element, a pumping chamber being arranged between this element and the remaining interior of the plastic sleeve and a nonreturn valve being provided between the pumping chamber and the remaining interior, this nonreturn valve closing when the element is depressed and opening when it returns. The pumping chamber communicates with a permanently open outlet opening in the head of the container. Arranged between the pumping chamber and the outlet opening is another nonreturn valve which opens when the element is depressed to release the adhesive and which closes when the element returns.

30       By applying pressure to the adhesive-filled container, it is possible to dispense the adhesive in precisely measured amounts.

The adhesive coatings are distinguished by rapid setting and excellent bond strength.

The claims  
CLAIMS

1. A ready-to-use adhesive pack, characterized by the following features in combination:
  - a) an adhesive based on emulsion polymers thickened to an ointment- or paste-like consistency which, although soft-spreading, will not flow in the absence of pressure and which contain an adhesive-type homo- and/or copolymer stabilized with protective colloids or thickeners as disperse solid in aqueous phase,
  - b) a hand-held container accommodating the adhesive with an outlet opening for the adhesive and a device for the measured release thereof,
  - c) a device which reduces the adhesive-filled interior of the container in volume commensurate with the release of the adhesive in such a way that the adhesive remaining in the container is always bubble-free and safe against the re-entry of air.
2. An adhesive pack as claimed in Claim 1, characterized in that stabilized aqueous emulsion polymers having viscosities (Brookfield RTV, 20°C/20 r.p.m.) of at least 15,000 mPas and preferably of at least 50,000 mPas are present as the adhesive, the viscosity of the thickened aqueous emulsion polymers best not exceeding 3,000,000 mPas and, more particularly, being in the range from 50,000 to 500,000 mPas.
3. An adhesive pack as claimed in Claim 1 or 2, characterized in that storable water-soluble and/or water-swellable polymer compounds, particularly of synthetic origin, are present as thickener in the adhesive.
4. An adhesive pack as claimed in Claim 1 or any of the following Claims, characterized in that the adhesive contains polyethylene glycol or mixed polyethers of ethylene oxide and propylene oxide having an average molecular weight of at least 3000 as thickeners having virtually unlimited stability in storage.

5. An adhesive pack as claimed in Claim 1 or any of the following Claims, characterized in that aqueous emulsion polymers having the following composition:

Polymer solids	approx. 20 to 70% by weight
preferably	35 to 60% by weight
Thickener	0.5 to 15% by weight
preferably	2 to 12% by weight
Water	approx. 30 to 75% by weight

and optionally containing further auxiliaries, such as emulsifiers, thixotropizing agents, water-miscible substantially involatile solvents, preservatives, plasticizers and the like, are present as the adhesive.

6. An adhesive pack as claimed in Claim 1 or any of the following Claims, characterized in that homo- and/or co-polymers based on vinyl esters, more especially vinylacetate, acrylic acid and/or methacrylic acid or derivatives thereof, more especially their esters and/or amides, or on dienes are present as the adhesive-type polymer.

7. An adhesive pack as claimed in Claim 1 or any of the following Claims, characterized in that ointment or paste-like aqueous polymer compositions which form films hardening by drying and/or permanently tacky, pressure-sensitive films are present as the adhesive.

8. An adhesive pack as claimed in Claim 1 or any of the following Claims, characterized in that polyethylene glycols having an average molecular weight of at least 5000 and preferably in the range of from about 12,000 to 20,000 are present as thickener.

9. An adhesive pack as claimed in Claim 8, characterized in that the polyethylene glycols are present in quantities of at least 3% by weight and preferably in quantities of from about 3 to 10% by weight, based on the total weight of the

emulsion.

10. An adhesive pack as claimed in Claim 8 or 9, characterized in that the polyethylene glycols are present in admixture with polyvinyl alcohol, at least substantially equivalent quantities of polyethylene glycol and polyvinyl alcohol being present, although the polyethylene glycol is best present in an excess of, in particular, about 1 to 2 parts by weight of polyethylene glycol to 1 part by weight of polyvinyl alcohol.
11. An adhesive pack as claimed in Claim 1 or any of the following Claims, characterized in that a distributing element is provided for spreading the adhesive leaving the outlet opening onto a surface to be bonded.
12. An adhesive pack as claimed in Claim 1 or any of the following Claims, characterized by a dispenser operable by the holding hand for dispensing the adhesive.
13. An adhesive pack as claimed in Claim 1 or any of the following Claims, characterized in that the handle is in the form of a cylindrical plastic sleeve (1;17;33), in the lower end of which a piston-like closure element (2) is slidably mounted in such a way that, under the effect of the vacuum produced inside the cylindrical plastic sleeve when the dispenser is operated, the closure element (2) moves automatically towards the dispenser at the head of the cylindrical plastic sleeve.
14. An adhesive pack as claimed in Claim 1 or any of the following Claims, characterized in that the outlet opening is in the form of a permanent opening (24;36) which is designed to be closed by means of a closure cap (25;37) which also surrounds the dispenser.
15. An adhesive pack as claimed in Claim 1 or any of the following Claims, characterized in that the container is in the form of a plastic or metal tube impermeable to the diffusion of water vapor.
16. An adhesive pack as claimed in Claim 1 or any of

Claims 2 to 12, characterized in that the container is in the form of a double-walled tube having an outer wall of an elastically deformable material and, accommodated therein, an air- and water-tight, permanently and inelastically deformable film bag for holding the adhesive and in that at least one vent opening is provided in the elastically deformable outer wall.

17. The use of emulsion polymers adjusted to an ointment- or paste-like consistency which, although soft-spreading, will not flow in the absence of pressure and which contain an adhesive-type homo- and/or copolymer stabilized with thickeners as disperse solid in aqueous phase, in handle-like plastic containers with a headpiece, comprising an outlet opening, a pressure-controlled dispense operable by the holding hand and a distributing element for the dispensed part of the emulsion polymer, as a ready-to-use adhesive pack held in one hand.

18. The use claimed in Claim 17, characterized in that polyethylene glycols having an average molecular weight of at least 3000 are used as stable, non-coagulating thickener.

19. The use claimed in Claims 17 and 18, characterized in that polyethylene glycols having an average molecular weight of at least 5000 to 35,000 and preferably of from about 12,000 to 20,000 are used.

20. The use claimed in Claims 17 to 19, characterized in that the polyethylene glycols are present in quantities of at least 3% by weight and preferably in quantities of from about 3 to 10% by weight, based on the total weight of the emulsion.

DATED this SEVENTEENTH day of JULY 1985

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Patent Attorneys for the Applicant  
SPRUSON & FERGUSON

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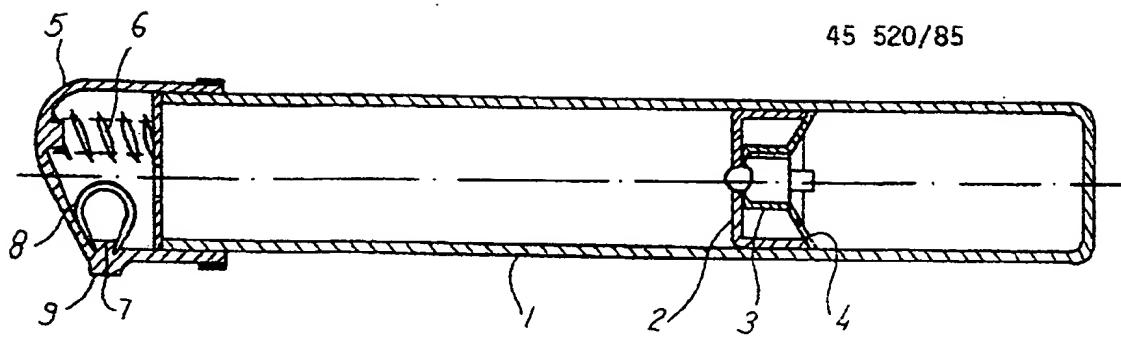


FIG. 1.

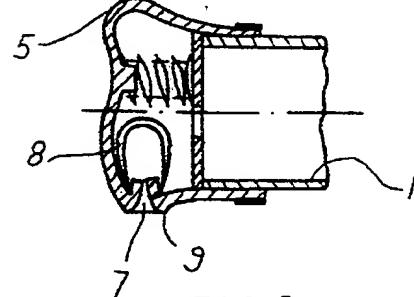


FIG. 2.

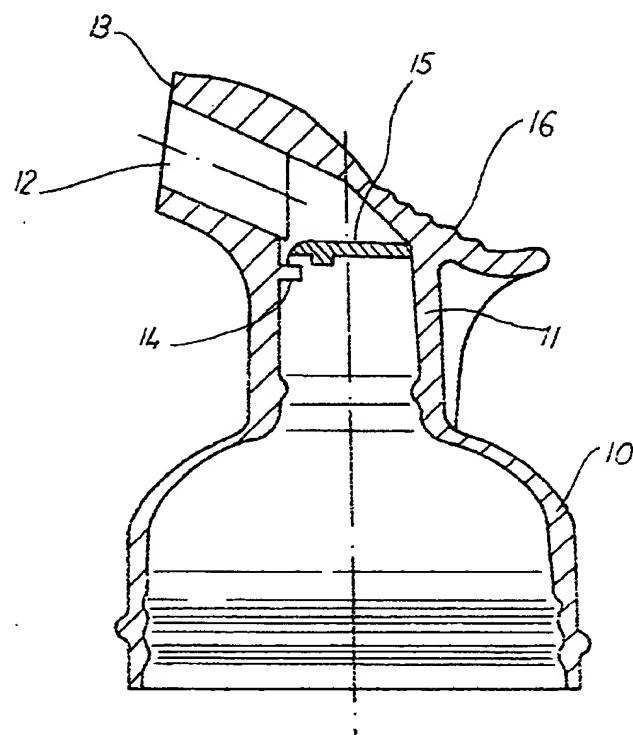


FIG. 3.

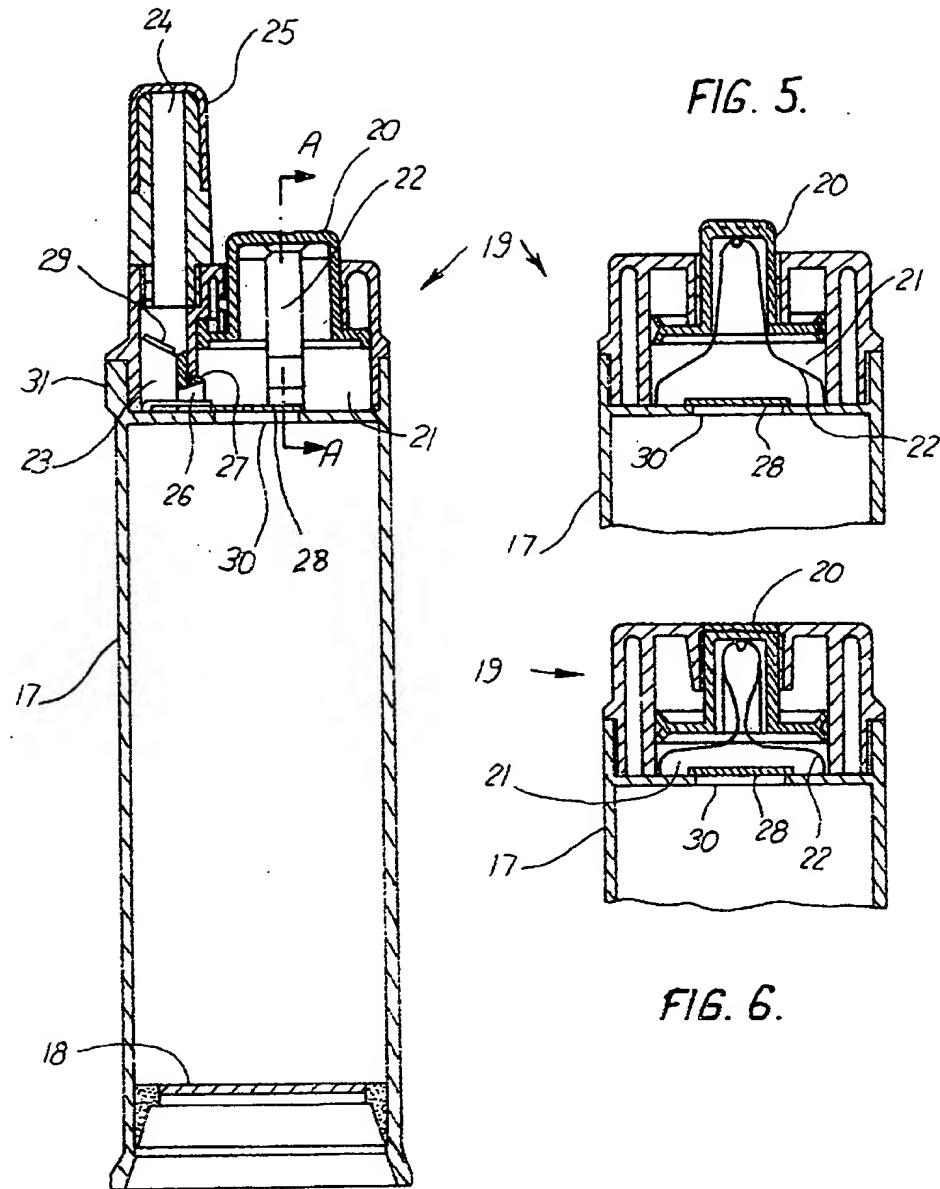


FIG. 4.

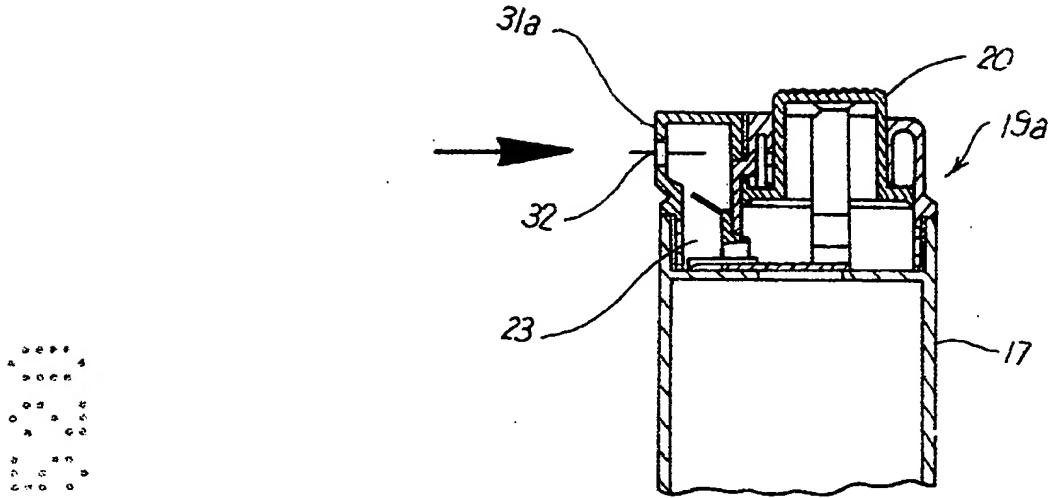


FIG. 7.

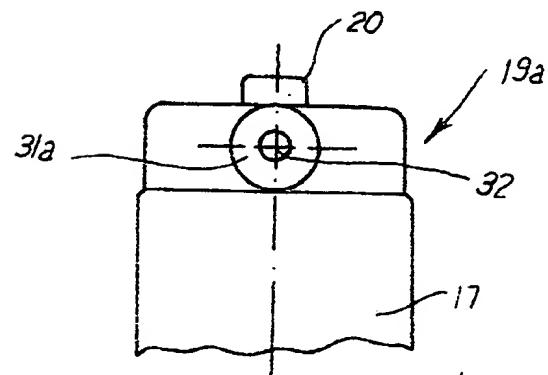


FIG. 8.

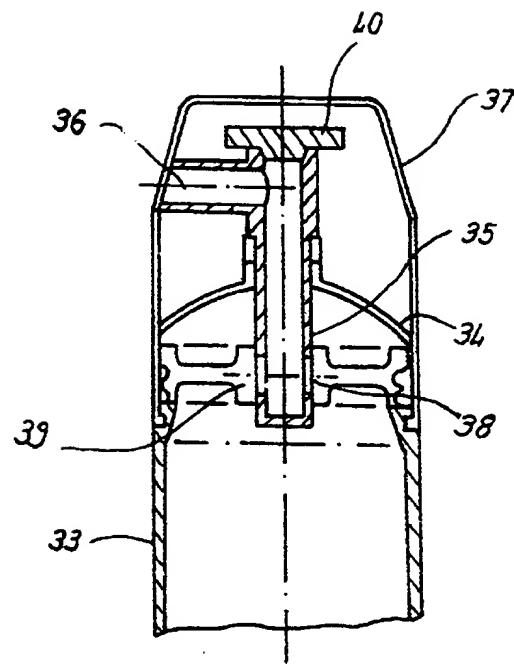


FIG. 9.

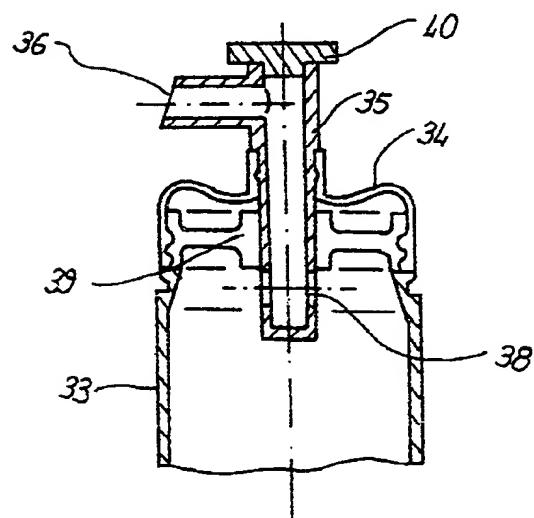


FIG. 10.

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